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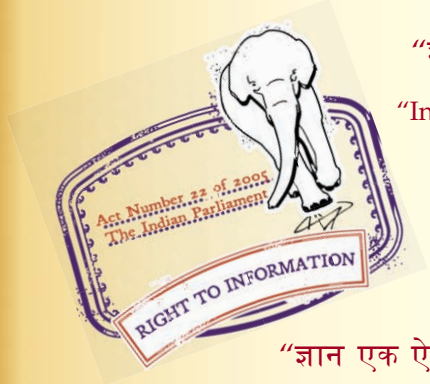
IS 7159 (1984): Methods of test for henna powder [PCD 19: Cosmetics]



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Indian Standard

METHODS OF TEST FOR HENNA POWDER

(First Revision)

UDC 665·58 : 633·863·8 : 543

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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April 1985

Indian Standard

METHODS OF TEST FOR HENNA POWDER

(First Revision)

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(*Continued on page* 2)

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(*Continued on page* 9)

Indian Standard

METHODS OF TEST FOR HENNA POWDER

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 13 December 1984, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This Indian Standard was first published in 1973. Over the years, based on the experience gained, the Sectional Committee decided to revise this standard and also prepare a separate product specification covering requirements of henna powder.

0.3 Adequate data are not available in literature on the physico-chemical characteristics of henna powder and its methods of analysis. The Sectional Committee has prepard this standard on the basis of available information and experience gained during analysis of samples of henna powder.

0.4 In this revision, additional tests have been included to minimize the chances of adulteration. Tests for quantitative estimation of colouring pigment lawsone in henna powder based on spectrophotometric analysis; determination of moisture and volatile matter and fineness have also been included.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard lays down the methods of test for henna powder.

*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the result of analysis.

3. PREPARATION OF SAMPLE FOR ANALYSIS

3.1 Procedure — If the sample is in coarse condition, grind and/or crush it as rapidly as possible without loss or grain of moisture and with minimal exposure so that it passes through a 250-micron sieve. Mix the sample thoroughly, divide and keep at least 100 g portion in a non-corrodible, clean and dry air-tight container for analysis.

4. DETERMINATION OF MOISTURE AND VOLATILE MATTER

4.1 Procedure — Weigh accurately about 5 g of the prepared sample material in a moisture *dish*, about 6 to 8 cm in diameter and about 2 to 4 cm in depth. Dry in an air oven at a temperature of 105 ± 2°C to constant mass (within ± 5 mg).

4.2 Calculation

$$\text{Moisture and volatile matter, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = loss in mass in g on drying, and

M = mass in g of the material taken for the test.

5. DETERMINATION OF COLD WATER EXTRACT

5.1 Procedure — Weigh to the nearest 0·001 g, about 2 g of the prepared sample. Transfer the material quantitatively with water to a 100-ml volumetric flask and fill to the mark with cold water. Stopper the flask and shake at approximately 30 minutes intervals for 8 hours and allow to settle for another 16 hours without shaking. Filter the extract through a dry filter paper. Reject first few millilitres, then evaporate a 25-ml aliquot to dryness in a tared dish on the water-bath and heat in the oven at 100 ± 2°C to constant mass. Record the final mass.

*Specification for water for general laboratory use (*second revision*).

5.2 Calculation

$$\text{Cold water soluble extract, percent by mass} = 4 \times \frac{M_2}{M_1} \times 100$$

where

M_1 = mass in g of the test sample, and

M_2 = mass in g of the residue obtained.

6. DETERMINATION OF CRUDE FIBRE

6.1 Reagents

6.1.1 *Petroleum Ether* — low boiling.

6.1.2 *Dilute Sulphuric Acid* — 1·25 percent (*m/v*), accurately prepared.

6.1.3 *Sodium Hydroxide Solution* — 1·25 percent (*m/v*), accurately prepared.

6.1.4 *Ethyl Alcohol* — 95 percent (*v/v*).

6.2 Procedure — Weigh accurately about 2·5 g of the prepared sample. Transfer the material into a one-litre flask. Take 200 ml of the dilute sulphuric acid in a beaker and bring to boil. Transfer the whole of the boiling acid to the flask containing material and immediately connect the flask with a water-cooled reflux condenser and heat, so that the contents of the flask begin to boil within 1 minute. Rotate the flask frequently, taking care to keep the material from remaining on the sides of the flask and out of contact with the acid. Continue boiling for exactly 30 minutes. Remove the flask and filter through fine linen (about 18 threads to the centimetre) or through a coarse acid-washed, hardened filter paper, held in a funnel, and wash with boiling water until the washings are no longer acid to litmus. Bring some quantity of sodium hydroxide solution to boil under a reflux condenser. Wash the residue on the filter into the flask with 200 ml of boiling sodium hydroxide solution. Immediately connect the flask with the reflux condenser and boil for exactly 30 minutes. Remove the flask and immediately filter through the linen or through filter paper. Thoroughly wash the residue with boiling water and transfer to a Gooch crucible prepared with a thin but compact layer of ignited asbestos. Wash the residue thoroughly first with hot water and then with about 15 ml of ethyl alcohol and with three successive washings of 15 ml of petroleum ether each. Dry the Gooch crucible and contents at 105 ± 1°C in an air-oven for 3 hours, cool and weigh. Repeat the process of drying for 30 minutes, cooling and weighing until the difference between two consecutive weighings is less than 1 mg. Incinerate the contents of the Gooch crucible in the muffle furnace at 550 ± 20°C until all the carbonaceous matter is burnt. Cool the Gooch crucible containing the ash in a desiccator and weigh.

6.3 Calculation

$$\text{Crude fibre, percent by mass} = \frac{100\,(M_1 - M_2)}{M}$$

where

M_1 = mass in g of Gooch crucible and contents before ashing,

M_2 = mass in g of Gooch crucible and contents after ashing, and

M = mass in g of the material taken for the test.

7. DETERMINATION OF MINERAL MATTER

7.1 Procedure — Weigh accurately about 5 g of the prepared sample in a silica dish. Heat the dish at first on a low flame and then in a muffle furnace maintained at about 600°C. Cool in a desiccator and weigh. Repeat the process of heating, cooling and weighing until constant mass is obtained. Preserve the ash for test under **8.**

7.2 Calculation

$$\text{Mineral matter, percent by mass} = \frac{M_2 \times 100}{M_1}$$

where

M_2 = mass in g of the ash, and

M_1 = mass in g of the material taken for the test.

8. DETERMINATION OF ACID INSOLUBLE ASH

8.1 Reagent

8.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

8.2 Procedure — To the ash preserved in **7.1** add 25 ml of dilute hydrochloric acid (5 N), heat on water-bath for 10 minutes, allow to cool and filter the contents of the dish through Whatman filter paper No. 42, wash the filter with distilled water till the washing is free from acid. Return the filter and residues to the dish. Keep it in oven to dry and ignite to free from carbon (in muffle furnace to 600°C). Cool the dish in desiccator and weigh. Repeat the process of igniting, cooling and weighing, until the difference between two successive weighings is less than one milligram. Note the lowest mass.

8.3 Calculation

$$\text{Acid insoluble ash, percent by mass} = \frac{M_2}{M_1} \times 100$$

where

M_2 = mass in g of the residue, and

M_1 = mass in g of the material taken for test in **7.1**.

9. DETERMINATION OF EXTRANEOUS SAND

9.1 Procedure — Weigh 5 g of prepared sample and transfer to a beaker. Add about 100 ml of carbon tetrachloride and mix the contents intimately and allow to settle. The supernatant liquid is decanted and the residue again stirred with another 100-ml portion of carbon tetrachloride. This process is repeated till it is freed of all vegetable matter. Transfer the residue to a tared silica dish, ignite and weigh to constant mass.

NOTE — That the residue has been freed from vegetable matter can be judged by the absence of formation of light leafy scum on inside of the beaker when carbon tetrachloride evaporates partially.

9.2 Calculation

$$\text{Extraneous sand, percent by mass} = \frac{M_2}{M_1} \times 100$$

where

M_2 = mass in g of sand, and

M_1 = mass in g of material taken for test.

10. THIN LAYER CHROMATOGRAPHIC (TLC) TEST FOR DETECTION OF EXTRANEOUS DYES

10.1 Preparation of Plates — Mix 10 g of the silica gel (of TLC grade, particle size 10 to 40 μm) with 20 ml of distilled water to make a slurry and spread over glass plates to a depth of 250 microns. Activate the plates for 30 minutes, by keeping in an oven maintained at 105°C.

10.2 Preparation of Sample — Extract about 0·1 g of the prepared sample with 5 ml of chloroform and apply one drop of the extract on the base line of the plate.

10.3 Development of Chromatogram — Keep the prepared plates in a jar containing a mixture of chloroform : methyl ethyl ketone : glacial acetic acid (5 : 4 : 1). Allow the solvent front to run up to 15 cm (the time taken is about 3 hours) at room temperature (25 to 30°C). Observe the spots obtained, under ultra violet light.

10.4 The principal ingredients of genuine henna powder, namely lawsone and chlorophyll will give spots on the chromatogram as given below:

Lawsone	Reddish spot with Rf value approximately 0·4
Chlorophyll	Greenish spot coincident with the liquid front

10.4.1 Any other spots on the chromatogram indicate extraneous dyes.

10.4.2 For comparison, use a reference sample prepared from standard henna powder.

11. DETERMINATION OF LAWSONE PIGMENT CONTENT

11.1 Outline of Method — The pigment is extracted and the lawsone content is determined by comparing the observed optical density (measured colorimetrically) with a calibration curve, relating optical density to various concentrations of 2-hydroxy, 1-4-naphthoquinone.

11.2 Apparatus

11.2.1 *Spectrophotometer or Photoelectric Colorimeter* — with a filter of 490 nm.

11.3 Reagents

11.3.1 *Sodium Bicarbonate Solution* — 5 percent (*m/v*).

11.3.2 *2-Hydroxy, 1-4-naphthoquinone*

11.4 Procedure

11.4.1 *Preparation of Standard Calibration Curve* — Construct a calibration curve by dissolving known amounts of 2-hydroxy, 1-4-naphthoquinone ($C_{10}H_6O_3$) varying in concentration from 0 to 2 percent under the same condition as described in **11.4.2** below.

11.4.2 Weigh 2·0 g of the prepared sample. Transfer it to a 100-ml volumetric flask. Add 5 percent (*m/v*) solution of sodium bicarbonate and make up the volume to mark. Shake the contents of the flask every half an hour or so for about 8 hours. Allow to settle overnight, thereafter filter the solution through a filter paper and reject the first few millilitres. Take 10 ml of the filtrate in a 25-ml volumetric flask and dilute with distilled water up to the mark. Measure the optical density of this solution with a spectrophotometer at 490 nm.

11.5 Calculation — Refer to the calibration curve and determine the percent lawsone content of the sample from the curve.

(*Continued from page* 2)

Ad-hoc Panel for Henna Powder, PCDC 19 : 1 : 2

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INDIAN STANDARDS

ON

COSMETIC RAW MATERIALS

IS:

263-1983 Boric acid for cosmetic industry (*fourth revision*)

918-1968 Calcium carbonate, precipitated, for cosmetic industry (*first revision*)

1462-1977 Talc for cosmetic industry (*second revision*)

1463-1977 Kaolin for cosmetic industry (*second revision*)

1767-1980 Dicalcium phosphate for dentifrice (*second revision*)

2519-1983 Calcium stearate for cosmetic industry (*second revision*)

2520-1977 Zinc stearate for cosmetic industry (*first revision*)

2521-1977 Magnesium stearate for cosmetic industry (*second revision*)

2528-1977 Magnesium carbonate for cosmetic industry (*second revision*)

2529-1983 Magnesium oxide for cosmetic industry (*second revision*)

2850-1983 Zinc oxide for cosmetic industry (*second revision*)

2851-1983 Titanium dioxide for cosmetic industry (*second revision*)

3986-1981 Sodium lauryl sulphate for cosmetic industry (*second revision*)

3987-1983 Sorbitol solution (70 percent) (*second revision*)

4028-1982 Beeswax, bleached, for cosmetic industry (*second revision*)

4236-1977 Glyceryl monostearate for cosmetic industry (*first revision*)

4652-1980 Ethyl *p*-hydroxybenzoate for cosmetic industry (*first revision*)

4653-1977 Methyl *p*-hydroxybenzoate for cosmetic industry (*first revision*)

4887-1980 Petroleum jelly for cosmetic industry (*first revision*)

5340-1981 Lanolin, anhydrous, for cosmetic industry (*first revision*)

5356-1983 *iso*-propyl myristate for cosmetic industry (*second revision*)

6333-1977 Propyl *p*-hydroxybenzoate for cosmetic industry (*first revision*)

6334-1980 Butyl *p*-hydroxybenzoate for cosmetic industry (*first revision*)

7101-1983 Coconut diethanolamide (*first revision*)

7299-1974 Mineral oil for cosmetic industry

9601-1980 Sodium silicate for cosmetic industry

9681-1980 Stearic acid for cosmetic industry

9830-1981 Water soluble sodium carboxy methyl cellulose for cosmetic industry

9831-1981 Sodium hydroxide for cosmetic industry

10283-1982 Ethyl acetate for cosmetic industry

10301-1982 *iso*-propyl alcohol for cosmetic industry

AMENDMENT NO. 1 APRIL 1990
TO
IS 7159 : 1984 METHODS OF TEST FOR HENNA POWDER

(First Revision)

(*Page* 7, *clause* **10.3**, *lines* 3 *and* 4) — Substitute ' (around 45 minutes) ' *for* ' (the time taken is about 3 hours) '.

(PCD 19)

Reprography Unit, BIS, New Delhi

AMENDMENT NO. 2 JULY 2004
TO
IS 7159 : 1984 METHODS OF TEST FOR HENNA POWDER

(First Revision)

(*Page* 8, *clause* **11.4**) — Substitute the following by the existing:

'**11.4 Procedure**

11.4.1 *Preparation of Standard Calibration Curve*

Prepare standard 0.1 percent stock solution of 2-hydroxy, 1-4-naphthoquinone ($C_{10}H_6O_3$) by dissolving it in sodium bicarbonate solution, 5 percent (*m/v*). Pipette 0 ml (Blank), 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of standard 0.1 percent stock solution in 100 ml volumetric flasks and dilute with sodium bicarbonate solution 5 percent, (*m/v*) up to the mark. Measure the optical density of these solutions having varying concentration from 0.0, 10, 20 to 50 ppm of 2-hydroxy, 1-4-naphthoquinone, at 490 nm with a spectrophotometer and construct a calibration curve. The stock solution should not be stored for more than one month.

11.4.2 Weigh 2.0 g of the prepared sample. Transfer it to a 100 ml volumetric flask. Add sodium bicarbonate solution, 5 percent (*m/v*) and make up the volume to mark. Shake the contents of the flask every half an hour or so for about 8 h. Allow to settle overnight. Thereafter filter the solution through a filter paper and reject the first few millilitres. Take 10 ml of the filtrate in a 25 ml volumetric flask and dilute with 5 percent sodium bicarbonate solution up to the mark. Further take 10 ml of this solution in a 100 ml volumetric flask and dilute with 5 percent sodium bicarbonate solution up to the mark. Measure the optical density of this solution with a spectrophotometer at 490 nm.'

(PCD 19)

Reprography Unit, BIS, New Delhi, India